

<Experimental report>

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<OBJECT>

An object of this experimental report is to assess the swelling degree of binder described in the examples 3 and 7 of U.S. Patent No. 6,756,153 ('153) in an electrolyte, whose component of monomer is in the range of the present application.

<EXPERIMENT>

1. Polymerization of monomer

(Example 1)

Example 1 is corresponding to examples 3 and 7 of '153.

To 250 parts of water, 70 parts of butyl acrylate, 10 parts of acrylic acid, 15 parts of methacrylic acid, 5 parts of 1,3-butadiene, 3 parts of Sodium dodecylbenzenesulfonate and 0.3 parts of ammonium persulfate were added, and polymerization was conducted in a polymerization vessel at 60°C for 8 hours. Then, the content was cooled to room temperature, and an aqueous 5% sodium hydroxide solution was added to adjust the pH to 7 whereby a pH-adjusted latex was obtained.

Next, N-methylpyrrolidone (hereinafter, it may be abbreviated to NMP) as an organic liquid dispersion medium was added to the pH-adjusted latex. The mixture was evaporated by an evaporator under a reduced pressure with an aspirator at 80°C in a water bath until the water content becomes 800ppm, whereby a dispersion in NMP of polymer particles having a solid concentration of 13% was obtained.

In example 3 of '153, (A) an ethylenically unsaturated monomer soluble in NMP is butyl acrylate, (B) an ethylenically unsaturated monomer insoluble in NMP is acrylic acid, methacrylic acid, and 1,3-butadiene.

(Example 2)

Example 2 is corresponding to example 1 of the present application.

Into an autoclave with a stirrer, 400 parts of ion-exchange water, 26 parts of acrylonitrile, 5 parts of sodium dodecylbenzene

sulfonate and 3 parts of potassium persulfate were charged, and then the solution was sufficiently stirred. Thereafter, the solution was heated to 60°C to initiate polymerization at a first stage. When the polymerization conversion ratio reached 85%, thereto were added 48 parts of 2-ethylhexyl acrylate as a monomer for a second stage to continue the reaction. When the polymerization conversion ratio at the second stage reached 90%, thereto were added 26 parts of acrylonitrile as a monomer for a third stage. When the polymerization conversion ratio reached 99%, the solution was cooled to a room temperature, and lithium hydroxide was added to adjust the pH thereof to 7, where by a pH-adjusted latex was obtained.

Next, NMP was added thereto, and then water therein was volatilized with an evaporator to yield a dispersion of a polymer in NMP, having a solid concentration of 8%.

2. A swelling degree in an electrolyte

Aliquid obtained by dissolving or dispersing 0.2 g of the polymer in 10 mL of NMP was subjected to flow casting on a sheet made of polytetrafluoroethylene, and then dried to obtain a cast film. 4 cm² area piece of the cast film was cut out, and the weight thereof was measured. Thereafter, the piece of the cast film was immersed into an electrolyte of 60°C temperature. The immersed film was picked up after 72 hours, and then the electrolyte adhering on the film surface was wiped with a paper towel. Immediately, the weight of the film was measured. The value of (the weight after the immersion)/(the weight before the immersion) was defined as the swelling degree. As the electrolyte, there was used an electrolyte obtained by dissolving LiPF₆ in the concentration of 1 mole/liter into a solvent of 1:2 (volume ratio at 20°C) mixture of ethylene carbonate (EC) and diethyl carbonate (DEC).

<RESULT>

The result is shown in Table-1 below.

(Table-1)

	a swelling degree in an electrolyte
Example 1	5.1
Example 2	1.7

<DISCUSSION>

It is obvious that examples described in '153, whose component of monomer is in the range of the present application, exhibit a swelling degree of more than 4 in an electrolyte.